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SYNTHETIC MAGNESIUM SILICATE HYDRATES

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SYNTHETIC MAGNESIUM SILICATE HYDRATES

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INVENTORS:

JULIE CHI-SUN YANG

[• Boolean](#)

OWNERS:

JOHNS-MANVILLE CORPORATION

[• Advanced](#)

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1 This invention relates to synthetic silicate composi-
2 tions and to the preparation and to the use thereof in
3 commercial applications. More particularly, this invention
4 relates to magnesium silicates or magnesium containing sili-
5 cate compositions having highly adsorptive characteristics.
6 Such materials are especially adapted in the refining of
7 liquids or solutions in which it is desired to remove an
8 impurity such as coloring matter whereby the silicate can
9 selectively sorb a particular impurity. More particularly,
10 the invention is concerned with a magnesium silicate composi-
11 tion capable of providing higher adsorptive capacity than
12 heretofore obtainable by commercial magnesium silicates.

13 Solid adsorptive compositions are used extensively in
14 removing coloring matter or other impurities such as fatty
15 acids in refining liquids or other solutions. A particular
16 use is in the treatment of petroleum fractions wherein
17 adsorbents are used to remove undesirable contaminants. The
18 art has long recognized that hydrated silicates including
19 magnesium silicates have particular adaptation for such
20 purifying. Its properties are such that it may be pulverized
21 and thus provide substantial surface area. Additionally it
22 is characterized by a remarkably high adsorbency. Accord-
23 ingly, it may be used in other applications such as removing
24 impurities from oils, fats and waxes, and it has also found
25 valuable application in selective adsorption of vitamins,
26 dyes and similar chemical compounds. To produce these
27 highly adsorptive silicates, several methods have been used
28 in the prior art.

29 A first method may be illustrated by United States
30 Patent No. 2,393,625 wherein a soluble magnesium salt is
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1 reacted with a soluble silicate. This is done in a concen-
2 trated slurry of the two constituents in order to precipitate
3 a magnesium silicate gel. In such a general process the
4 temperature of the reacting solution is generally strictly
5 controlled so as to prevent flocculation. Accordingly, a
6 temperature above 150°F. is avoided. In the practice of such
7 a process, magnesium compounds such as magnesium acetate,
8 magnesium chloride, magnesium sulphate and other similar
9 soluble magnesium salts have been successfully used. Like-
10 wise, a soluble silicate is employed in order to provide a
11 suitable source for the silica radical. Exemplary of such a
12 soluble silicate are compounds such as sodium silicate. Such
13 strict temperature control is maintained substantially
14 throughout the remainder of the process, except that the gel
15 so formed is later subjected to high temperature conditions.
16 But this is specifically after the reaction is complete and
17 the chemical and physical nature of the silicate determined.
18 Thus the heating is substantially nothing more than a drying
19 step.

20 A second method of providing synthetic magnesium silicate
21 compositions is illustrated by United States Patent Nos.
22 1,999,210, 2,173,085 and 2,241,791. These patents generally
23 depict a process of hydrothermally producing hydrous sili-
24 cates. The reaction is somewhat similar to that of the pre-
25 cipitated process explained above in that the starting
26 materials may be the same. However, the choice of the initial
27 materials is not limited to those materials possessing
28 complete water solubility. In general, this method provides
29 that the constituents are mixed together in a slurry,
30 suspension or solution of water and the reaction carried out
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1 under hydrothermal conditions; i.e., elevated temperatures
2 and pressures are used in order that the constituents may
3 hydrothermally react.

4 All these prior teachings suggest that a ratio of mag-
5 nesia to silica of about 0.5 produces satisfactory results
6 and represents the type of product obtainable by all such
7 ratios. The prior teachings have recognized that synthetic
8 magnesium silicates have good adsorptive characteristics.
9 However, in many industrial applications, it has been
10 determined that these particular products do not possess an
11 adsorption capacity as high as would be desired.

12 It is believed that this low adsorption capacity is
13 directly related to the amount of available surface area.
14 Accordingly, if additional surface area can be provided, more
15 adsorption may be realized with a given quantity of material.
16 However, the prior art does not teach how this increase in
17 surface area may be obtained. Accordingly, it was believed
18 that all the products produced under the varying reaction
19 conditions possess substantially the same surface area.
20 Further, the art has not provided adsorbents wherein a
21 particular removal efficiency may be altered, i.e., increased
22 or decreased, without deleteriously affecting its ability
23 to remove other contaminants.

24 These products should also possess other characteristics
25 such as light weight, chemical inertness, fine and porous
26 structure, etc., which will allow continued use as the previ-
27 ous synthetic silicates, i.e., as fillers, abrasives, rein-
28 forcing agents in chemical compositions such as synthetic
29 plastics, etc.

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1 Accordingly, it is an object of this invention to
2 provide an improved magnesium silicate composition possessing
3 adsorptive properties greater than those heretofore obtain-
4 able.

5 It is another object of this invention to provide a
6 synthetic magnesium silicate which may be produced from the
7 same constituents as those previously used in the art with
8 new and improved characteristics.

9 It is another object of this invention to disclose and
10 provide a magnesium silicate possessing unique adsorptive
11 characteristics due to increased surface area.

12 It is still another object of this invention to disclose
13 and provide a substantially chemically inert product in
14 finely divided form which will render it useful as an adsorp-
15 tive agent but also possessing other characteristics such as
16 lightweight, fine and porous structure, etc.

17 It is yet another object of this invention to disclose a
18 method in producing silicates of magnesium having improved
19 adsorptive characteristics.

20 Another object of this invention is to disclose a method
21 of providing magnesium silicates having highly developed
22 surface area and highly porous structure and is particularly
23 advantageous for adsorptive purposes.

24 A still further object of this invention is to provide
25 operating conditions such as mol ratios, temperatures, pres-
26 sures, drying conditions, whereby a more effective form of
27 hydrated silicates may be provided.

28 Other objects and further scope of this invention as may
29 hereinafter appear will be readily apparent to those skilled
30 in the art. However, it should be understood that the
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1 following detailed description while indicating preferred
2 embodiments of the invention is given by way of illustration
3 only, since various changes and modifications within the
4 scope and spirit of the invention are readily apparent.

5 Since it is thought that increased surface area would
6 correspondingly increase the adsorptive capacity, a study was
7 made to determine how the surface area may be increased.

8 In all mol ratio compositions formed within the prior
9 art temperatures, which are generally substantially below
10 450°F., the normal structure of the silicate is amorphous
11 and foil shaped. Nevertheless, the amorphous agglomeration
12 is characterized by a good adsorptive capacity. Additionally,
13 it is known that at temperatures above 450°F., the magnesium
14 silicate is crystalline in form, and accordingly has less
15 surface area. The very nature of crystalline material is
16 that it is satisfied within its structure and is hence stable
17 and less subject to react.

18 Since crystalline products were not as desirable due to
19 the low adsorptive capacity, it was previously theorized that
20 a commercially acceptable product produced under the most
21 economical conditions would be quite desirable. Thus, only
22 the temperature and pressure necessary to produce an
23 amorphous silicate were used.

24 It has now been discovered that generally speaking, at
25 the transition point, i.e., just prior to conversion to
26 crystals, the compositions are unstable and accordingly very
27 reactive. However, as will be explained below, this does not
28 prove uniform for all mol ratios of the initial constituents.

29 In general, the method of this invention may be carried
30 out according to the previously disclosed processes; that is,
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1 a slurry or suspension of a magnesia containing material may
2 be formed and the silica providing constituent added either
3 as a powder, paste or slurry. The mixture is then subjected
4 to the application of heat for time sufficient to permit the
5 hydrothermal reaction to reach substantial completion. It
6 has now been found that by carefully controlling the mol
7 ratio of K_2O to SiO_2 and the temperature and condition of the
8 reactions, products superior to those heretofore obtainable
9 may be produced.

10 Particular control of the reacting conditions is
11 important due to the following.

12 The microscopic structure of the normal magnesium sili-
13 cate of the prior art possesses a standard surface area by
14 applying some heat and pressure. A new physical structure,
15 crystalline in nature, is obtained wherein extreme heat and
16 pressure are applied. It is discovered as one approaches the
17 transition point where the finely divided amorphous powder
18 changes to the crystalline structure, the surface of the
19 particles can be greatly enhanced. The transition point is,
20 of course, dependent upon the particular mol ratio employed.
21 However, it is generally about $350^{\circ}F.$ to about $450^{\circ}F.$ Above
22 this transition temperature the product has a more defined
23 crystalline structure and the particles are large in size.
24 These particles are well stacked thin sheets or minute rods
25 depending on the initial composition. It is therefore clear
26 that high temperature results in a pronounced decrease in
27 surface adsorbing ability of the products. At temperatures
28 below $400^{\circ}F.$, well crystallized phases cannot be found. At
29 such temperatures, the magnesium content facilitated the
30 formation of a crumbled floccular structure, its quality
31 depending on the amount of the magnesium content. Accord-
32 ingly, the magnesium oxide-silica-water system must stay at tem-
33 peratures below $450^{\circ}F.$ in order to get the maximum surface area.

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1 It was observed that by controlling the amount of pure
2 starting material under various hydrothermal conditions, two
3 distinct phases and consequential combinations of these two
4 may be formed. One of these phases was similar to the
5 mineral talc wherein the MgO/SiO_2 of ratio is approximately
6 0.5. On the other hand, where ratios on the order of 1.5
7 were employed, a product quite similar to chrysotile was
8 formed.

9 Therefore, in the preliminary group of reactions mol
10 ratios of 0.5 to 1.5 were employed. The temperature was con-
11 trolled in order to substantially correspond to the transition
12 temperature and accordingly ranged between $300^{\circ}F.$ and $450^{\circ}F.$
13 The corresponding pressure is generally determined by the
14 particular temperature chosen, as it is desirable to use a
15 saturated steam system. However, super-or subsaturated
16 systems are operable.

17 It was discovered that when the ratio was maintained at
18 0.5 and the temperature and pressure raised, the amorphous
19 form was transformed directly into the crystalline form. This
20 is thought to be so because of the platy nature of the initial
21 composition thereby readily convertible to sheet form. On
22 the other hand, wherein higher ratios are employed, the
23 initial composition's physical structure broke down prior to
24 becoming crystalline. It is in this physical state or transi-
25 tion stage that the composition is most unstable. Accordingly
26 it is quite susceptible to reacting, and further the surface
27 area is substantially increased. From light photomicrograph
28 observations it was found that rather complete reactions took
29 place under these conditions. Almost no unreacted silica
30 providing material could be detected in the product with a
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1 high magnesium content and fewer reacted silica diatoms were
2 found in samples having a normal chemical composition. All
3 of the products produced and especially the high magnesium
4 compositions were very fine in size and easily dispersed.
5 The electron photomicrograph evidenced that the magnesium
6 silica hydrates had a crumbled foil form. The following are
7 examples of particular reactions to illustrate the above
8 process.

9 EXAMPLE I

10 A diatomaceous earth sold under the trademark CELITE and
11 a magnesium hydroxide filter cake were used as raw materials.
12 Various samples of the materials were each reacted in a
13 5-gallon reactor for various time intervals ranging from about
14 3 hours to about 1 week. And the temperature was varied
15 between about 300°F. and about 450°F. It had been previously
16 recognized that the surface area of hydrated silicate
17 products may be appreciably increased by a subsequent acid
18 treatment. Accordingly, some of the samples were treated by
19 adding to the reacted slurry 1 gram of concentrated sulfuric
20 acid per 4 grams of solid at 160°F. to 180°F. for approxi-
21 mately 1 hour before filtering and drying. The mol ratio was
22 varied between 0.5 and 1.5. The maximum surface areas
23 obtained at an MgO/SiO₂ ratio of 1.5 was 284 square meters
24 per gram. This is compared with the average area of 195 m²/g
25 for an 0.5 ratio.

26 The hydrothermal synthetic silicates of various MgO/SiO₂
27 ratios are tabulated in Table I below. All resultant products
28 were obtained after filtering, washing and draining. The
29 surface area measurements were made by a gas adsorption
30 specific surface area test known as the BET method. The
31 results of these tests are also shown in Table I.

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TABLE 1

Sample	MgO/SiO ₂ Ratio	Condition for Synthesis	Acid Treat- ment	Surface Area m ² /g
1	0.5	300°F, 53 psi, 4 hr.	no	-
2	0.5	350°F, 100 psi, 3 hr.	yes	169.9
3	0.5	350°F, 100 psi, 3 hr.	no	164.7
4	0.5	450°F, 410 psi, 4 hr.	yes	249.8
5	0.75	392°F, 210 psi, 3.5 hr.	yes	223.2
6	0.75	392°F, 210 psi, 3.5 hr.	no	-
7	0.75	540°F, 950 psi, 1 wk.	yes	145.2
8	0.75	540°F, 950 psi, 1 wk.	no	190.0
9	1.5	300°F, 53 psi, 4 hr.	no	227.6
10	1.5	350°F, 100 psi, 3 hr.	yes	134.8
11	1.5	350°F, 100 psi, 3 hr.	no	203.3
12	1.5	392°F, 210 psi, 3.5 hr.	yes	284.1
13	1.5	392°F, 210 psi, 3.5 hr.	no	-
14	1.5	450°F, 410 psi, 4 hr.	yes	247.8

EXAMPLE II

A pilot plant reaction in a 375-gallon reactor was made using the following reaction conditions:

MgO/SiO₂ molar ratio 0.85
 Reaction temperature 385-390°F.
 Reaction time 3.5 hr.
 Reacted slurry solids content 0.93 lb./gal.
 Acid treatment 25 lb. H₂SO₄
 100 lb.
 solids

The reactants, magnesium hydroxide and diatomaceous earth were mixed together in a water slurry and were added to a preheated reactor. At the end of the reaction period, the product slurry was drained from the reactor through a cooler into a tank. Sulfuric acid (66°Ba) was added to the slurry (at 150 - 160°F.) and was allowed to react for one hour before filtering, washing and drying. The dried product was pulverized through a 0.020 harrington screen in the No. 1 Micropulverizer. The physical properties of this product, are summarized in Table 2 as Sample 1. The surface area of this product was 364 square meters per gram.

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TABLE 2

Physical Properties of Magnesium Silicate Batches

Sample No.	1	2
MgO/SiO ₂ Ratio	0.85	0.75
Reaction Time, hours	3.5	3.5
Reaction Temperature	390	410 - 415
Acid Treatment, %	25	25
Physical Properties		
Loose Weight, per	12.4	11.3
Water Absorption, %	155	-
Oil Absorption, %	-	166
Surface Area, sq m/g	364	376
% Moisture	9.8	7.0
Total % Ignition Loss at 1800°F.	19.5	12.9
pH	7.3	6.9

EXAMPLE III

A series of small-scale reactions were made to determine the effect of variations in mol ratio, reaction temperature and reaction time on the product surface area.

The ranges investigated were:

MgO/SiO₂ mol ratio 0.65 - 0.95
 Reaction temperature 360 - 450°F.
 Reaction time 2 hr. and 3.5 hr.

The results obtained are summarized in Table 3 below. These results indicated that the 0.75 mol ratio would be the most promising in the 390 - 450°F. temperature range. But it is significant to note that all products produced within the transition range were far superior to those previously used (approximately 195 m²/g). A 3.5 hour pilot plant reaction was made with a MgO/SiO₂ mol ratio of 0.75 at 415°F. The reacted slurry was treated with 1 pound of 66°Be sulfuric acid per 4 pounds of solids. The physical properties of this product after filtration, drying, and pulverizing are listed in Table 2 as Sample 2. The product surface area was 376 square meters per gram.

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TABLE 3

Reaction Temperature °F.	Reaction Time Hours	Product Mol Ratio			
		0.65	0.75	0.85	0.95
360	2.0	-	375	-	-
	3.5	-	115	330	-
390	2.0	405	365	-	350
	3.5	380	115	320	375
420	2.0	385	370	330	345
	3.5	380	450	365	345
450	2.0	300	420	360	-
	3.5	390	435	385	-

Various tests to determine the effectiveness of the color removal and the fatty acid removal were performed by checking 1 gram of the sample with 100 ml. of liquid. The results are shown in Table 4 wherein the data is presented in percentages of the original color solution or fatty acid addition. Of course, it is highly desirable to remove as much of the contaminant as possible but acceptable percentages for color and acid removal depend on the intended use. It can be observed from the Table that test samples X and Y which represent the former material sold as CELKATE T-21 by Johns-Manville are inferior in both color and fatty acid removal.

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TABLE 4

Test Sample	MgO/SiO ₂ Ratio	Condition for Synthesis	Acid Treat-ment	Color Removal %	Fatty Acid Removal %
A	3/2	350°F, 120 psi, 3 hr.	yes	70.3	23.6
B	3/2	Same	no	64.3	23.6
C	3/2	300°F, 53 psi, 4 hr.	yes	67.6	21.6
D	0.5	350°F, 120 psi, 3 hr.	yes	70.5	21.6
E	0.5	Same	no	65.8	33.5
F	0.5	450°F, 410 psi, 4 hr.	yes	82.0	19.7
G	0.5	Same	no	72.3	21.6
H	3/4	540°F, 950 psi, 1 wk.	no	54.7	17.7
I	3/4	392°F, 210 psi, 3.5 hr.	yes	71.3	21.6
J	3/2	392°F, 210 psi, 3.5 hr.	yes	73.4	43.0
X	0.5	350°F, 120 psi, 2-4 hr.	yes	61.9	14.4
Y	0.5	350°F, 120 psi, 2-4 hr.	yes	61.8	17.7

It was concluded that from the above test that various adsorptive qualities may be obtained, all of which are higher than those previously known by carefully controlling the mol ratio and the reacting conditions.

Generally speaking, the greater the value of the surface area of the compound, the higher the adsorbing ability and thus the more efficient it is in fatty acid and color removal. For materials with a fixed MgO/SiO₂ ratio of about 0.5, the value of the surface areas for samples prepared for 450°F. was the greatest. This decreased when the temperature was lowered to 350°F. For samples with a higher magnesium content, that is, a MgO/SiO₂ of about 1.5, the value of the surface area reached a maximum when the sample was prepared at 350°F. For samples prepared under 450°F., increase of magnesium content of the starting material generally resulted in a product having a higher surface area.

In general, the acid treatment increased the surface area of most of the magnesium silicate hydrates. No differences between the treated and non-treated materials could be

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1 observed by electron microscope examinations. A study was
2 made of the actual effect of sulfuric acid on the surface
3 area. Samples of the reacted slurry taken before pilot
4 plant acid treatment were acid treated in a laboratory at
5 160°F. to 180°F. for about 1 hour. The amounts of concen-
6 trated sulfuric acid of the specific gravity of 1.84 were
7 varied and the surface areas of the various products are
8 shown in Table 5.

TABLE 5

Grams H_2SO_4	Surface Area
100 g. solids	sq. meters/g.
10	394
20	412
30	254
40	218

14 One additional test used to determine the effectiveness of
15 acid treated adsorbents is as follows:

16 This test is basically a contact adsorption method where
17 1% of an adsorbent is placed in a 0.2% potassium hydroxide
18 solution that was heated to 95°C. A nitrogen atmosphere is
19 required for the test. The amount of potassium hydroxide
20 removed is the index to the efficiency of the adsorbent. The
21 particular standard chosen is dependent on the actual amount
22 of efficiency required. For purposes of this test a 70%
23 standard was chosen. Samples of various acid treated
24 materials and their efficiency in this particular test are
25 shown in Table 6.

TABLE 6

% Acid	Surface Area	Efficiency
10	394	62
20	412	64
30	254	72
40	218	78

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1 These particular results indicated that under certain appli-
2 cations maximum surface area is not necessarily the control-
3 ling factor in good adsorption. A sample was taken of the
4 prior art material which would correspond to an 0.5 ratio
5 and lower hydrothermal conditions and it was accordingly
6 acid treated to determine its effective qualities under the
7 same test. The results are shown in Table Y.

TABLE Y

% Additional Acid	Efficiency
20	46
30	52
40	68
50	65

13 It is seen that a maximum removal is obtained at 40% treat-
14 ment and is below the selected 70% standard.

15 It was found that for fatty acid removal the new composi-
16 tions which contained high magnesium content were far
17 superior to samples of the previously recognized adsorbents,
18 i.e., those which contained about a 0.5 mol ratio. These
19 materials were far superior not only in adsorbing ability
20 but also in the filtration. A product developed which had a
21 mol ratio of 1.5 and was prepared at 392°F., 210 psi for
22 about 3-1/2 hours exhibited fatty acid removal about three
23 times as effective as the previous material.

24 It is accordingly concluded that the production of
25 synthetic magnesium silicates does not result in the same
26 product when the reaction conditions are varied. The data
27 presented herein shows that by carefully controlling the
28 reaction conditions so as to substantially fall in the
29 transition range and also by maintaining a fairly high
30 magnesia content, the adsorptive effectiveness of magnesium
31 silicates is greatly enhanced.

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1 With regard to the operable starting materials, the
2 only necessary requirement is that they be capable of
3 supplying magnesia and silica. Of course, the ease by which
4 the requirement is met and relative costs play an important
5 role in the actual determination. Accordingly, any of the
6 raw materials previously used are operable for purposes of
7 this invention. Such magnesia supplying constituents are
8 magnesium oxides, hydroxides, carbonates, sulfates, citrates,
9 acetates, chlorides, etc. Silica may be supplied by any
10 soluble silicates, diatomaceous earth, silica gel, silicic
11 acid, etc.

12 It is to be understood that the term "transition point"
13 as used herein and in the appended claims means that point
14 substantially immediately prior to which the amorphous form
15 is converted to a crystalline structure.

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THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A process of making silicates of high adsorption capacity comprising the steps of hydrothermally reacting magnesia and silica in a mol ratio of between 0.75 and 1.5 under such heat and pressure as to correspond to that point substantially immediately prior to which the amorphous form of magnesium silicate is converted to a crystalline structure for the particular mol ratio.
2. A process of making silicates of high adsorption capacity comprising the steps of hydrothermally reacting magnesia and silica magnesia in a mol ratio of between 0.75 and 1.5 at a temperature between 300 and 450°F.
3. A process as described in claim 1 wherein the reaction is conducted for such a period of time as to substantially completely react the initial components.
4. A method as described in claim 2 wherein the reaction is conducted between 2 and 4.5 hours.
5. An article of manufacture particularly characterized by adsorption capacity and good decolorization comprising a magnesium silicate containing magnesia and silica in the mol ratio between 0.75 and 1.5 produced under hydrothermal conditions corresponding to that point substantially immediately prior to which the amorphous form of magnesium silicate is converted to a crystalline structure of said silicate.

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6. An article of manufacture particularly characterized by adsorption capacity and good decolorization comprising an acid treated magnesium silicate containing magnesia and silica in the mol ratio between 0.75 and 1.5 produced under hydrothermal conditions corresponding to that point substantially immediately prior to which the amorphous form of magnesium silicate is converted to a crystalline structure for the mol ratio.

7. An article in manufacturing characterized by high adsorption capacity comprising a magnesium silicate containing magnesia and silica in a mol ratio of 0.85.

8. An article in manufacturing characterized by high adsorption capacity comprising a magnesium silicate containing magnesia and silica in a mol ratio of 0.85 formed under hydrothermal conditions corresponding to that point substantially immediately prior to which the amorphous form of magnesium silicate is converted to a crystalline structure.

9. An article which is described in claim 8 wherein a temperature of 390°F. is employed.

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